(Me$_2$Si), 22.8 (CH$_3$), 91.8 (CH$_2$=C), 127.9, 129.7, 133.3, 137.7 (phenyl ring carbons), 155.8 (C=CH$_2$). Anal. Calcd for $C_{11}H_{16}OSi_2$: C, 68.69; H, 8.38. Found: C, 68.46; H, 8.35.

Photolysis of 16 in the Presence of 2,3-Dimethylbutadiene. A mixture of 0.9011 g (4.10 mmol) of **16**, 1.5650 g (19.1 mmol) of 2,3-dimethylbutadiene, and 0.0386 g (0.250 mmol) of undecane as an internal standard in 120 mL of hexane was photolyzed for 2 h. The mixture was analyzed by GLC as being **20** (4% yield), **23** (20% yield), and **24** (42% yield), in addition to 23% of the starting compound **16**. Products **20**, **23**, and **24** were isolated by preparative GLC. For **23**: MS *m/e* 302 (M$^+$); IR 1427, 1247, 1011, 833 cm^{-1}; ^{1}H NMR δ 0.07 (s, 6 H, Me$_2$Si), 0.33 (s, 6 H, Me$_2$Si), 1.57 (br s, 9 H, three CH$_3$), 1.90 (s, 2 H, CH$_2$), 6.69 (s, 2 H, HC(Si)=CH(Si)), 7.30–7.53 (m, 5 H, phenyl ring protons); ^{13}C NMR δ −2.7 (Me$_2$Si), −2.6 (Me$_2$Si), 20.4 (CH$_2$), 21.2 (CH$_3$), 24.5 (two CH$_3$), 121.0 (C_a=C_b), 127.8 (C_b=C_a), 127.7, 128.7, 133.9, 138.7 (phenyl ring carbons), 148.3 (C_aH=C_bH), 152.4 (C_bH=C_aH). Anal. Calcd for $C_{18}H_{30}Si_2$: C, 71.45; H, 9.99. Found: C, 71.16; H, 9.99. For **24**: MS *m/e* 302 (M$^+$); IR 1426, 1428, 1159, 1132 cm^{-1}; ^{1}H NMR δ −0.04 (s, 6 H, Me$_2$Si). 0.27 (s, 6 H, Me$_2$Si), 0.42–0.61 (m, 4 H, CH$_2$CH$_2$), 1.79 (s, 2 H, CH$_2$), 1.99 (s, 3 H, CH$_3$), 4.75 (br s, 1 H, HC=C), 4.95–5.03 (m, 3 H, H$_2$C=C and HC=C), 7.32–7.58 (m, 5 H, phenyl ring protons); ^{13}C NMR δ −3.6 (Me$_2$Si), −3.5 (Me$_2$Si), 7.6 (CH$_2$), 7.9 (CH$_2$), 21.0 (CH$_3$), 21.9 (CH$_2$), 110.2 (CH$_2$=C), 113.4 (CH$_2$=C), 127.7, 128.7, 132.9, 133.6 (phenyl ring carbons), 145.6 (two C=CH$_2$). Anal. Calcd for $C_{18}H_{30}Si_2$: C, 71.45; C, 9.99. Found: C, 71.37; H, 10.00.

Acknowledgment. This research was supported in part by a Grant-in-Aid for Scientific Research on Priority Area of Organic Unusual Valency, No. 02247104, from the Ministry of Education, Science, and Culture, to which our thanks are due. We also express our appreciation to Shin-Etsu Co. Ltd., Nitto Electric Industrial Co. Ltd., and Dow Corning Japan Ltd. for financial support.

Oxidative-Addition Reactions of Alkyl Chlorides with Chlorobis(cyclooctene)rhodium(I) and the Bis(oxazolinyl)pyridine Ligand: Formation of Stable (Chloromethyl)rhodium(III) Complexes and Their Reactions

Hisao Nishiyama,* Mihoko Horihata, Tsuyoshi Hirai, Shigeru Wakamatsu, and Kenji Itoh

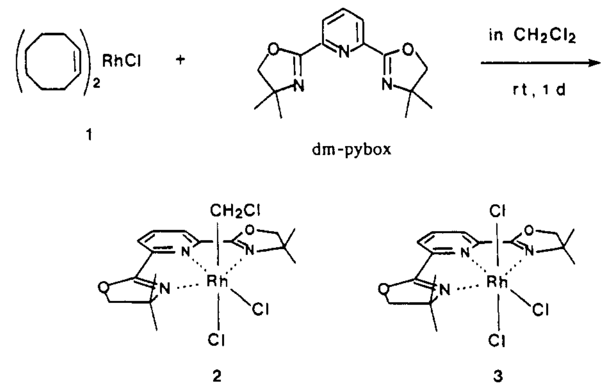
School of Materials Science, Toyohashi University of Technology, Tempaku-cho, Toyohashi 441, Japan

Received February 27, 1991

Dichloromethane and chloroform readily react with chlorobis(cyclooctene)rhodium(I) (1) and 2,6-bis(4,4-dimethyloxazolin-2-yl)pyridine, dm-pybox, at room temperature to give the stable (chloromethyl)rhodium(III) complex 2 and (dichloromethyl)rhodium(III) complex 4, respectively. Several organic halides also react with 1 to give the corresponding substituted alkylrhodium(III) complexes. Some reactions of the chloromethyl complexes were also demonstrated. Decomposition of 2 and 3 in methanol at 100–130 °C gave the methylrhodium(III) complex, in which the origin of the methyl group can be traced to be methanol.

We have prepared new terdentate nitrogen ligands, bis(oxazolinyl)pyridine (pybox), for asymmetric hydrosilylation of ketones with rhodium catalysts.[1] During the study of the pybox–rhodium complexes as catalysts, we have unexpectedly found an oxidative addition reaction of dichloromethane and chloroform to chlorobis(cyclooctene)rhodium(I) in the presence of the pybox, giving (chloromethyl)rhodium(III) complexes. Halomethyl transition-metal complexes have been well investigated from the aspect of the activation of halogenomethanes and the intermediates of catalytic systems.[2] We report here the formation and some reactions of the stable (chloromethyl)- and (dichloromethyl)rhodium(III) pybox complexes.

Scheme I



Results and Discussion

A mixture of chlorobis(cyclooctene)rhodium(I) (1)[3] and bis(4,4-dimethyloxazolin-2-yl)pyridine,[1b] dm-pybox, in

---

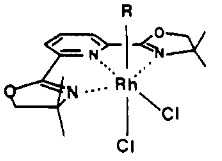
(1) (a) Nishiyama, H.; Sakaguchi, H.; Nakamura, T.; Horihata, M.; Kondo, M.; Itoh, K. *Organometallics*, 1989, *8*, 846. (b) Nishiyama, H.; Kondo, M.; Nakamura, T.; Itoh, K. *Ibid.* 1991, *10*, 500.

(2) (a) Collman, J. P.; Murphy, D. W.; Dolcetti, G. *J. Am. Chem. Soc.* 1973, *95*, 2687. (b) Werner, H.; Paul, W.; Feser, R.; Zolk, R.; Thometzek, P. *Chem. Ber.* 1985, *118*, 261. (c) Werner, H.; Hofmann, L.; Feser, R.; Paul, W. *J. Organomet. Chem.* 1985, *281*, 317. (d) Marder, T. B.; Fultz, W. C.; Calabrese, J. C.; Harlow, R. L.; Milstein, D. *J. Chem. Soc., Chem. Commun.* 1987, 1543. (e) Moss, J. R.; Peeling, S. *J. Organomet. Chem.* 1982, *236*, 221. (f) Weinberger, B.; Tanguy, G.; DesAbbayes, H. *Ibid.* 1985, *280*, C31. (g) Huser, M.; Youinou, M.-T.; Osborn, J. A. *Angew. Chem., Int. Ed. Engl.* 1989, *28*, 1386. (h) Alcock, N. W.; Pringle, P. G.; Bergamini, P.; Sostero, S.; Traverso, O. *J. Chem. Soc., Dalton Trans.* 1990, 1553. (i) Ghilardi, C. A.; Midollini, S.; Moneti, S.; Orlandini, A.; Scapacci, G.; Traversi, A. *Ibid.* 1990, 2293.

(3) van der Ent, A.; Onderdelinden, A. L. *Inorg. Synth.* 1973, *14*, 92. $(C_2H_4)_2RhCl_2$ could also be utilized for the oxidative addition.

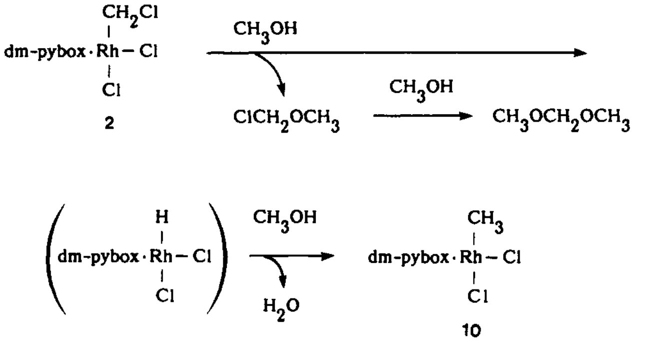
0276-7333/91/2310-2706$02.50/0

Scheme II



	-R
4	$-CHCl_2$
5	$-CH_2C_6H_5$
6	$-CH_2CH=CH_2$
7	$-CH_2COOMe$
8	$-CH_2CN$
9	$-CH_2COC_6H_5$
10	$-CH_3$

Scheme III



dichloromethane was stirred at room temperature under nitrogen for 1 day to give orange precipitates, which were collected by filtration and purified by column chromatography to give the stable (chloromethyl)rhodium complex, *cis*-Cl_2(axial-$ClCH_2$)Rh^{III}(dm-pybox) (**2**) (89%) accompanied with the trichloride **3** (4%). The stereochemistry of complex **2** was readily established on the basis of the unsymmetrical ^1H and ^{13}C NMR patterns of the signals.

Chloroform also reacts with the rhodium(I) complex **1** yielding the stable (dichloromethyl)rhodium complex **4** (47%) and the trichloride complex **3** (30%). Carbon tetrachloride gave only the trichloride **3** (95%), whose formation may indicate that a one-electron path for the oxidative addition dominates, depending on the nature of the organic halides, as reported before.[2a,4] Oxidative addition reactions with other organic chlorides, benzyl chloride, allyl chloride, $ClCH_2COOCH_3$, $ClCH_2CN$, and $ClCH_2COPh$, were examined and gave the corresponding axial-alkyl complexes **5**–**9**.[5]

We have then examined some reactions of the chloromethyl complex **2**. The decomposition of **2** in methanol at 100 °C for 1 day resulted in formation of the (axial-methyl)Rh complex **10** in 83% yield and trichloride **3** (15%).[6] The origin of the methyl group of **10** proved to be methanol as the solvent, since the reaction with methanol-d_4 exhibited the complete loss of the doublet signal at δ 1.25 ppm of the (methyl)Rh protons on the ^1H NMR spectrum. Furthermore, stoichiometric formation of dimethoxymethane was also detected by GC and ^1H NMR analysis.[7] We think that the decomposition could be initiated by nucleophilic attack of the methanol on the chloromethyl carbon atom, as previously reported for the several (halomethyl)metal species (Scheme III).[2b,c,e,f] The hydride atom on the unstable intermediary hydride–rhodium species **11** could act as an acidic proton, which causes dehydrative methylation, giving the methyl complex **10**.

In contrast, decomposition of **2** in ethanol at 100 °C predominantly gave the trichloride **3** (>95%) accompanied with diethoxymethane (>90%). The intermediary ethyl species might be so unstable to react readily with hydrogen chloride, giving the trichloride.

Formation of the methyl complex **10** was also confirmed by heating the Cl_2CHRh complex **4** and the $PhCH_2Rh$ complex **5** in methanol, respectively.[8]

The authentic axial-methyl complex **10** was obtained in 86% yield by methathesis of the trichloride **3** with $AlMe_3$ (1.5 equiv) at 0 °C.[9]

Some other reactions of **2** and **3** were as follows: Reduction of the trichloride **3** with sodium amalgam gave metallic rhodium, and the dm-pybox ligand was recovered in 71%. The reduction of **3** by using $LiAlH_2(OCH_2CH_2OCH_3)_2$ in CH_2Cl_2 gives the chloromethyl complex **2** (34%), which indicates the formation of the unstable monovalent rhodium species with dm-pybox followed by oxidative addition of CH_2Cl_2.

We have thus disclosed the oxidative addition of dichloromethane and chloroform, which seldom give stable oxidative adducts,[4] in particular by using nitrogen ligand pybox. Characterization and some reactions of the product (alkyl)rhodium complexes were demonstrated.

Experimental Section

General Considerations. All reactions were carried out under an atmosphere of nitrogen. Tetrahydrofuran was distilled from sodium. Dichloromethane and chloroform were distilled under nitrogen with $CaCl_2$ after being washed with water. ^1H (270 MHz) and ^{13}C (67.8 MHz) NMR spectra were recorded on a JEOL JNM-GX 270 spectrometer using tetramethylsilane as the internal reference. Infrared spectra were recorded on a JASCO A-3 spectrometer. GLPC analysis was performed with a Shimadzu GC-8A. Microanalyses were performed with Yanagimoto CHN corder MT-3. Analytical TLC was performed on Merck (Art 5715) precoated silica gel plates (0.25 mm). Column chromatography was performed with silica gel (Merck Art 7734) and silanized silica gel (Merck Art 7719). Other organic chemicals were used without any purification after purchased.

***cis*-Dichloro(chloromethyl)[2,6-bis(4,4-dimethyloxazolin-2-yl)pyridine]rhodium(III) (2).** A mixture of chlorobis(cyclooctene)rhodium (**1**) (359 mg, 1.0 mmol) and 2,6-bis(4,4-dimethyloxazolin-2-yl)pyridine (273 mg, 1.0 mmol), dm-pybox, in dichloromethane (10 mL) was stirred at room temperature under nitrogen in the dark for 1 day to give orange precipitates, which were collected by filtration to give the chloromethyl complex **2** (421 mg, 0.85 mmol). From the filtrate 20 mg (0.04 mmol) of **2** and 20 mg (0.04 mmol) of the trichloride **3** were obtained by column chromatography with CH_2Cl_2–MeOH as eluent; **2** included a trace amount of **3**. **2**: orange solids, mp >270 °C dec. ^1H NMR ($CDCl_3$): δ 1.63 (s, 6 H, CH_3), 1.70 (s, 6 H, CH_3), 4.67 (s, 4 H, O–CH_2), 4.96 (d, J_{Rh-H} = 3.2 Hz, 2 H, Rh–CH_2Cl), 7.86 (d, J = 7.8 Hz, 2 H, pyridine), 8.15 (t, J = 7.8 Hz, 1 H, pyridine). ^{13}C NMR ($CDCl_3$): δ 26.97, 27.69, 38.87 (d, J_{Rh-C} = 25.4 Hz, Rh–CH2Cl), 69.25, 84.09, 125.46, 138.17, 146.80, 166.13. IR (KBr): 1608 (O—C=N) cm^{-1}. R_f = 0.2 (EtOAc:MeOH = 5:1). Anal. Calcd for $C_{16}H_{21}N_3O_2Cl_3Rh$: C, 38.70; H, 4.26; N, 8.46. Found: C, 38.73; H, 4.21; N, 8.60. A crystal of **2** obtained by recrystallization from methanol was subjected to X-ray single-crystal

---

(4) Collman, J. P.; Hegedus, L. S.; Norton, J. R.; Finke, R. G. *Principles and Applications of Organotransition Metal Chemistry*, 2nd ed.; University Science Books: Mill Valley, CA, 1987; p 279.

(5) In THF (4 mL), **1** (1.0 mmol), and organic halide (5.0 mmol) at 0–20 °C, for several hours. Isolated yields: for benzyl chloride, **5** (38%) and **3** (31%); for allyl chloride, **6** (45%) and **3** (44%); for $ClCH_2COOCH_3$, **7** (79%) and **3** (5%); for $ClCH_2CN$, **8** (89%) and **3** (6%); for $ClCH_2COPh$, **9** (56%) and **3** (20%).

(6) Kinetics for the decomposition of **2** in methanol: first order for **2**, $k = 5.72 \times 10^{-5}\ s^{-1}$ at 378 °K, E_a = 29.3 kcal/mol.

(7) ^1H NMR for CH_3O–CH_2–OCH_3: (CD_3OD) δ 3.32 (CH_3), 4.54 (CH_2) ppm.

(8) From **4** at 80 °C for 20 h, **10** (20%) and **3** (74%); from **6** at 150 °C for 2 days, **10** (27%) and **3** (27%). The trichloride **3** itself does not decompose in methanol at 100–150 °C in a sealed tube.

(9) The axial-methyl complex **10** was also obtained in 34% yield by oxidative addition at room temperature for 3 days with an excess of CH_3Cl, dm-pybox, and **1** in THF in a sealed tube.

2708 *Organometallics* **1991**, *10*, 2708–2717

analysis but decomposed upon initial irradiation. For the X-ray analysis of the similar pybox–rhodium complex could be referred to ref 1b. For 3, see also ref 1b.

***cis*-Dichloro(dichloromethyl)[2,6-bis(4,4-dimethyloxazolin-2-yl)pyridine]rhodium(III) (4).** A mixture of **1** (131 mg, 0.36 mmol) and dm-pybox (100 mg, 0.36 mmol) in chloroform (3.6 mL) was stirred at room temperature under nitrogen in the dark for 1 day. The mixture was concentrated under reduced pressure, and the residue was purified by column chromatography with CH_2Cl_2–MeOH as eluent to give 81 mg (0.15 mmol) of the dichloromethyl complex **4** (42%) and 52 mg (0.11 mmol) of **3** (30%). The corresponding equatorial isomer was not detected. **4**: orange solids, mp 210 °C dec. ^1H NMR ($CDCl_3$): δ 1.75 (s, 6 H, CH_3), 1.80 (s, 6 H, CH_3), 4.59 (d, J = 8.6 Hz, 2 H), 4.67 (d, J = 8.6 Hz, 2 H), 7.46 (d, J_{Rh-H} = 3.4 Hz, 1 H, Rh-$CHCl_2$), 7.94 (d, J = 8.1 Hz, 2 H, pyridine), 8.22 (t, J = 8.1 Hz, 1 H, pyridine). ^{13}C NMR ($CDCl_3$): δ 26.45, 28.74, 69.88, 70.22 (d, J_{Rh-C} = 34.2 Hz, Rh–CH_2Cl), 84.07, 125.75, 139.05, 147.32, 164.84. IR (KBr): 1605 (O—C=N) cm^{-1}. R_f = 0.45 (EtOAc:MeOH = 5:1). Anal. Calcd for $C_{16}H_{21}N_3O_2Cl_3Rh{\cdot}0.5H_2O$): C, 35.58; H, 3.92; N, 7.78. Found: C, 35.53; H, 3.81; N, 8.20.

***cis*-Dichloro(methyl)[2,6-bis(4,4-dimethyloxazolin-2-yl)pyridine]rhodium(III) (10).** In a sealed tube, a solution of **2** (200 mg, 4.0 mmol) in methanol (10 mL) was heated at 100 °C for 2 days. After concentration the residue was purified by column chromatography with CH_2Cl_2–MeOH to give 155 mg (0.34 mmol) of the methyl complex **10** (83%) and 32 mg (0.06 mmol) of **3** (15%). **10**: orange solids, mp 250 °C dec. ^1H NMR ($CDCl_3$): δ 1.25 (d, J_{Rh-H} = 2.4 Hz, 3 H, CH_3–Rh), 1.65 (s, 6 H, CH_3), 1.86 (s, 6 H, CH_3), 4.61 (d, J = 13.5 Hz, 2 H), 4.65 (d, J = 13.5 Hz, 2 H), 7.84 (d, J = 7.8 Hz, 1 H, pyridine), 8.12 (t, J = 7.8 Hz, 2 H, pyridine). ^{13}C NMR ($CDCl_3$): δ 3.44 (d, J_{Rh-C} = 19.5 Hz, CH_3–Rh), 27.42, 27.47, 69.17, 84.05, 125.23, 137.29, 146.94, 163.66. IR (KBr): 1610 (O—C=N) cm^{-1}. R_f = 0.3 (CH_2Cl_2:MeOH = 5:1). Anal. Calcd for $C_{16}H_{22}N_3O_2Cl_2Rh$: C, 41.58; H, 4.80; N, 9.09. Found: C, 41.54; H, 5.13; N, 8.41.

Methylation of 3 with Trimethylaluminum. To a suspension of **3** (100 mg, 0.21 mmol) in dichloromethane (4.0 mL) was added a hexane solution of trimethylaluminum (0.36 mmol, 1.2 N, 0.30 mL) at 0 °C. The mixture was stirred to be homogeneous for 10 min and was then quenched with water (2 mL). The organic layer was collected, concentrated, and purified by column chromatography with CH_2Cl_2–MeOH to give 82 mg (0.18 mmol, 86%) of the methyl complex **10**.

Supplementary Material Available: Textual presentation of spectroscopic characterization data for **5–9** (2 pages). Ordering information is given on any current masthead page.

Binding of π-Acid Ligands in Diiridium and Rhodium–Iridium Iodo Complexes, Including Rare Examples of Ethylene Coordination in "A-Frame" Compounds. Structure of $[Ir_2I_2(CO)(\mu\text{-}CO)(Ph_2PCH_2PPh_2)_2]{\cdot}CH_2Cl_2$

Brian A. Vaartstra, Jianliang Xiao, James A. Jenkins, Robert Verhagen, and Martin Cowie*

Chemistry Department, University of Alberta, Edmonton, Alberta, Canada T6G 2G2

Received February 22, 1991

The diiodo species $[MIrI_2(CO)(\mu\text{-}CO)(dppm)_2]$ (M = Ir (**1**), Rh (**3**), dppm = $Ph_2PCH_2PPh_2$) are prepared by the reaction of the corresponding chloro complexes, $[MIrCl_2(CO)_2(dppm)_2]$, with KI. Abstraction of one iodide ion in **1** and **3**, with use of $AgBF_4$, yields the corresponding A-frame complexes $[MIr(CO)_2(\mu\text{-}I)(dppm)_2][BF_4]$ (M = Ir (**2**), Rh (**4**)). All four species react with CO to give tricarbonyl complexes, but further CO addition is not observed. Reaction of **1** with dimethyl acetylenedicarboxylate (DMAD) yields four isomers of $[Ir_2I_2(CO)_2(\mu\text{-}DMAD)(dppm)_2]$, although upon heating of this mixture three of these species transform into the fourth, in which each metal has one iodo and one carbonyl ligand attached, having a structure much like that previously determined for the dichloro analogue. Reaction of the cationic species **2** and **4** with DMAD yields $[MIr(CO)_2(\mu\text{-}I)(\mu\text{-}DMAD)(dppm)_2][BF_4]$, and reaction of the mixed-metal product with CO yields $[RhIrI(CO)_2(\mu\text{-}CO)(\mu\text{-}DMAD)(dppm)_2][BF_4]$, which displays unusually high ^{13}C–^{13}C coupling of 47.8 Hz between the bridging carbonyl and the terminally bound carbonyl on Ir. Compounds **1** and **2** react with ethylene to yield the unusual ethylene adducts $[Ir_2(CO)(C_2H_4)(\mu\text{-}I)(\mu\text{-}CO)(dppm)_2][X]$ (X^- = I^-, BF_4^-), in which ethylene is terminally bound to one iridium center. The structure of **1** has been determined by X-ray techniques. This compound crystallizes, as the CH_2Cl_2 solvate, in the space group $P2_1/c$ with a = 20.241 (4) Å, b = 14.153 (2) Å, c = 20.446 (2) Å, β = 112.76 (1)°, V = 5400.7 Å^3, and Z = 4 and has refined to R = 0.041 and R_w = 0.049 on the basis of 5949 observations and 346 parameters varied. This complex has the unusual structure in which one carbonyl is terminally bound and one is bridging. One iodo ligand is opposite the Ir–Ir bond on one metal, while the other iodo group is cis to the Ir–Ir bond on the adjacent metal. Both carbonyl groups are mutually cis.

Introduction

Much of our understanding about oxidative-addition reactions in mononuclear complexes is derived from studies of Vaska's compound, *trans*-$[IrCl(CO)(PPh_3)_2]$, and related species.[1-12] It is well-known, for example, that substitution of the chloride by the better π-donor iodide ligand results in an increase in the rate of oxidative addition of H_2[6,9] and

---

(1) Collman, J. P. *Acc. Chem. Res.* **1968**, *1*, 136.

(2) Vaska, L. *Acc. Chem. Res.* **1968**, *1*, 335.

(3) Collman, J. P.; Roper, W. R. *Adv. Organomet. Chem.* **1968**, *7*, 53.

(4) Halpern, J. *Acc. Chem. Res.* **1970**, *3*, 386.

(5) Collman, J. P.; Hegedus, L. S.; Norton, J. R.; Finke, R. G. *Principles and Applications of Organotransition Metal Chemistry*; University Science Books: Mill Valley, CA, 1987.

(6) Chock, P. B.; Halpern, J. *J. Am. Chem. Soc.* **1966**, *88*, 3511.

(7) Collman, J. P.; Kang, J. W. *J. Am. Chem. Soc.* **1967**, *89*, 844.

(8) Collman, J. P.; Sears, C. T., Jr. *Inorg. Chem.* **1968**, *7*, 27.

(9) Blake, D. M.; Kubota, M. *Inorg. Chem.* **1970**, *9*, 989.

(10) Uqo, R.; Pasini, A.; Fusi, A.; Cenini, S. *J. Am. Chem. Soc.* **1972**, *94*, 7364.

(11) Kubota, M.; Kifer, G. W.; Ishikawa, R. M.; Bencaia, K. E. *Inorg. Chem. Acta* **1973**, *7*, 195.

(12) Rees, W. M.; Churchill, M. R.; Li, Y.-J.; Atwood, J. D. *Organometallics* **1985**, *4*, 1162.

(13) McGinnety, J. A.; Doedens, R. J.; Ibers, J. A. *Science* **1967**, *155*, 709.

(14) Vaska, L. *Acc. Chem. Res.* **1976**, *9*, 175.

(15) Sutherland, B. R.; Cowie, M. *Inorg. Chem.* **1984**, *23*, 2324.

(16) Hutton, A. T.; Pringle, P. G.; Shaw, B. L. *Organometallics* **1983**, *2*, 1889.

(17) Vaartstra, B. A.; Cowie, M. *Inorg. Chem.* **1989**, *28*, 3138.

(18) Sutherland, B. R.; Cowie, M. *Organometallics* **1984**, *3*, 1869.

(19) Sutherland, B. R.; Cowie, M. *Organometallics* **1985**, *4*, 1801.

0276-7333/91/2310-2708$02.50/0